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NASA TN D-2264

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by Robert L. Summers

Lewis Research Center
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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • MARCH 1964

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SUMMARY

A study has been conducted to determine the effects of cesium vapor on ionization gages. Although no permanent damage to the gages was noted over a period of months, temporary gage failure due to cesium plating on the gage was noted after a few hours at cesium pressures above 10^{-6} torr.

At pressures below 10^{-6} torr, the response of the gage to changes in cesium pressure was inhibited by the condensation of cesium on the envelope and tubulation. For stable pressure readings from the gage, periods of several hours were required.

The ionization-gage sensitivity to cesium was 13.7 times the gage air sensitivity, with a probable error of 14 percent.

INTRODUCTION

The high-vacuum facilities used at this laboratory for ion- and electric-propulsion research require pressure gages with which the system pressures may be monitored. During the course of propulsion studies, pressure measurements are made when condensable metallic vapors, such as cesium, may be present in the vacuum space.

The sensitivity of any given ionization-gage design depends on the species of gas present within the vacuum space (refs. 1 and 2). Values of sensitivities for ionization gages have been published for many gases; however, there is no information available concerning the use of the ionization gage in the presence of cesium vapor. It was the purpose of this study to generate this information.

For the most part, this study deals only with commercial gages of the Bayard-Alpert type, run under conditions specified by the manufacturer.

IONIZATION-GAGE SENSITIVITY

The empirical equations governing ionization-gage operation are

$$i_+ = S_A p i_-$$

$$i_+ = r_{A/B} S_B p i_-$$

$$r_{A/B} = \frac{S_A}{S_B}$$

where

i_+ measured ion current, amp

i_- measured ionizing electron current, amp

p pressure within gage, torr

S_A, S_B gage sensitivity to gases A and B, respectively, $torr^{-1}$

The subscripts are, where convenient, the chemical symbols of the gases in question.

The ionization-gage sensitivity S is known to vary with gage geometry, electrical parameters, and the gas species present (refs. 1 and 2). The theories developed for the ionization gage (refs. 3 and 4) are accurate in predicting S to slightly more than a factor of 2.

It is reported (refs. 4 and 5) that apparently identical gages could give variations in indicated pressure of about 20 percent, about the same variation found between dissimilar gages. For pressure measurements with an accuracy better than 20 percent (ref. 6), individual gage calibration for each gas encountered is a necessity.

For this study, the accuracy desired was at least 20 percent. Also, because of the sensitivity dependence on electrical parameters, the parameters used were those recommended by the manufacturer.

APPARATUS AND PROCEDURE

The apparatus is shown in figures 1 to 3.

In the apparatus of figure 1, high-purity cesium metal is inserted into and sealed within an ionization gage under high-vacuum conditions. This apparatus is of all-glass construction and is capable of withstanding bakeout at 350°C. The cesium was prepared by reduction of cesium chloride with calcium metal in a nickel or tantalum bucket.

Because of the extreme chemical activity of cesium, the apparatus was baked at a temperature of 350° C and purged with argon prior to reduction of the cesium metal. System bakeout at this temperature caused no reaction within the cesium chloride - calcium mixture.

Subsequent elevation of the bucket temperature to approximately 600° C gave an easily controlled reaction of the cesium chloride - calcium mixture, which resulted in the evolution of high-purity cesium metal. The heat source was a tungsten filament mounted directly in the reduction bucket.

The cesium evolved condensed in the system directly above the gage tubulation (fig. 1). The wall temperature was held above the cesium melting point (approximately 30° C), so that the cesium condensed as a liquid and ran into the gage. Normally, 1 cubic centimeter of cesium was inserted in the gage in this manner. The ionization gage was then sealed and removed from the pumping system. In subsequent tests, the gage was mounted with the envelope above the tubulation (fig. 2). With the gage in this position, the cesium metal was driven into the tubulation by heating the envelope and cooling the tubulation.

During preliminary studies, a marked effect on gage sensitivity caused by cesium purity was observed. For control purposes, it was necessary to derive a technique to monitor this purity. With the method of gage preparation used, the only probable cesium contamination after the gage was sealed was oxygen from the gage envelope and from the glass-metal seals produced by chemical interaction with the cesium. Unpublished data from other investigators indicate that oxygen contamination of cesium causes a freezing-point depression of 1° C for 0.6 percent oxygen content. The melting point of cesium was assumed to be 28.49°±0.05° C. Cesium purity was determined by immersing the entire gage in a suitable water bath and observing the bath temperature at which the cesium melted. By a very slow change in the temperature, agitation of the immersed gage, and observation of the state of the cesium, repeated measurements of freezing points could be made to about ±0.1° C, which corresponded to an uncertainty of less than ±0.1 percent oxygen content. Since this test assumed the presence of oxygen only, care was taken to remove as many sources of contamination as possible in the preparation of the gage. The purity of the cesium was tested in this manner both before the tests and at frequent intervals during the course of the tests.

After preparation and purity tests, the ionization gage was mounted, with the sealed gage tubulation containing cesium, in the controlled temperature bath (figs. 2 and 3). This bath was controlled by thermoelectric heat pumps and a servoloop, which provided a temperature control of ±0.5° C at the outer faces of the bath block with no detectable temperature fluctuation in the bath proper. The range of controlled temperatures with this bath was -20° to 60° C.

Figure 3 shows the general arrangement of instrumentation used with the apparatus of figure 2 for control and collection of data. The gage control and measurement system was either a commercially available control unit or an equivalent system of power supplies and meters. The cesium vapor pressure within the gage was determined from the bath temperature, which varied less than ±0.1° C over the bath volume. Cesium vapor pressure P was calculated from the bath temperature T by the following equation from reference 7:

$$\log_{10}P = 10.546 - 4150/T - \log_{10}T \quad \text{for} \quad T < 302^\circ \text{ K}$$

with an accuracy of 1 percent over the range 220° to 350° K.

The tests, for the most part, were conducted with the gage envelope temperature established by the normal heat balance between radiation from the filament and convection to the environment. This temperature was about 50° C. The cesium temperature in the tubulation was not raised above 25° C during the course of the experiments.

When condensables such as cesium are used, the vapor pressure, after a period of time for stabilization, is determined by the point in the system of lowest temperature. For this reason and for prevention of excessive pumping of cesium into the gage, the gage temperature must always be in excess of the sealed tubulation temperature.

Preliminary tests showed that the only effect of changes in envelope temperature was an anticipated effect caused by transpiration.

In the performance of tests, the bath temperature was varied in steps, equilibrium conditions were established, and measurements were made. At the end of a test cycle, the tubulation was cooled to -20° C, and the gage envelope was heated by grid outgassing. This process drove the cesium from the gage into the tubulation. After the gage was allowed to cool, the original gage characteristics and background pressure were recovered.

The indicated background pressure within various gages with the techniques just described ranged from 5×10^{-8} to 5×10^{-7} torr. The background pressure within a given gage was relatively constant over the course of the study. The range of cesium vapor pressures used for data was 1×10^{-8} to 5×10^{-7} torr.

RESULTS

In the course of this investigation, several effects of cesium on the ionization gage were observed. These effects are inherent in the operation of ionization gages in the presence of cesium. The effects were all caused by the condensable nature of cesium. Anticipated effects observed were a lengthy time constant of the gage in response to a step pressure change, electrical leakage between elements, and photoelectric currents from the grid and the collector elements.

It was also noted that any slight contact with envelope caused large shifts in gage output. It is assumed that this effect was caused by a capacitive effect between a partial coating of cesium on the interior of the gage and the external environment. Reference 8 reports wide variations in gage sensitivity and attributes them to variations in envelope equilibrium potential. This effect, as well as photoelectric effects due to ambient lighting, was removed by enclosing the gage in a suitable blackened container.

Other effects that could not be removed were surface leakage, photoelectric currents due to the filament, and the gage lag in response to pressure changes.

The time lag, or time constant, of various gage configurations and conditions was observed. The observed time constant for a clean RG-75-type gage was about 1 to 2 hours. This figure varied with gage condition and configuration by

a factor of 10. Conditions that proved to be important were envelope surface area, tubulation diameter and length, and previous gage history of exposure to cesium vapors.

The various time-lag tests indicated that cesium was chemisorped on the gage envelope to a thickness of approximately 10 monolayers before any appreciable reevaporation of the cesium occurred. In addition, the effective tubulation conductance was considerably reduced until complete coating with cesium occurred. By these concepts, a calculation was performed that indicated a reasonable agreement with experiment. A typical gage response to a change in cesium pressure is shown in figure 4. During the measurement cycle shown, leakage currents were insignificant. This was verified by removing the filament heating current frequently and observing the various electrode currents with the biasing voltages applied.

The leakage currents were such as to cause failure of gages of the RG-75 type after about 1 hour of operation at a cesium vapor pressure of 10^{-6} torr. At lower pressures, of course, longer periods of satisfactory operation were possible.

In gages using an internally shielded collector lead, collector leakage was not observed until such time as the gage was inoperative because of grid-to-filament leakage currents through the condensed cesium surface film.

The effects of cesium on the gage, which have been discussed previously, were all highly dependent on the previous history of exposure of the gage to cesium vapor. The effects of air on a gage previously exposed to cesium vapor were not considered in this study.

Observations of the effects of cesium on gage elements were also made. Over a period of several months, no deterioration of the thoriated-iridium filament, used in some of the gage samples, was detected. Cesium, however, completely destroyed the platinum film elements of gages of the VG-1 type. Although consistent sensitivity measurements were obtained with the VG-1 gage, the platinum collector element soon disappeared. With the exception of gages with the platinum elements, no permanent gage damage was observed. Original gage characteristics could be recovered at any time by thorough outgassing of the gage.

Measurement of Gage Sensitivity

The gages used to determine gage sensitivity to cesium were two of the RG-75 Bayard-Alpert gages and one VG-1 triode gage. The RG-75 gages were operated at an emission current of 10 milliamperes, the VG-1 at 5 milliamperes. For purposes of calculation, gage air sensitivities were assumed to be those quoted by the manufacturer. The variations of the individual gages from the quoted sensitivities were less than 10 percent.

Typical measurements of ionization gage sensitivity are shown in table I and figure 5. The measurements were made in the following manner. With the gage tubulation containing cesium cooled to a very low temperature, a measurement of the collector residual or background current was made. This current was, for the

most part, caused by the residual gases in the gage at the time the gage was sealed. For measurement of the background current, the lower limit of the apparatus bath temperature (-20° C) was sufficiently low since cesium vapor pressure at this temperature is less than 10^{-9} torr.

The cesium was heated to some given temperature, equilibrium was established, and measurements were made. Cesium vapor pressure was determined at each new temperature from the equation in reference 7. The ion collector current was recorded, as well as the ionizing electron current. Minor variations in electron current were corrected by normalizing the ion current to a normal electron current of 10 milliamperes (5 ma for the VG-1 gage). By subtraction of the background current from each of the measured ion currents, the change in ion current, in response to changes in cesium pressure, was determined. From this, the gage sensitivity to cesium S_{Cs} could be calculated as well as the ratio of sensitivities r_{Cs}/air.

This technique was used since the background current remained relatively constant over a series of tests. After a cycle of the bath temperature, however, when the cesium pressure was returned to essentially zero, the gage output stabilized to a somewhat higher value of background current than was observed before the test cycle. This increase in collector current was nominally about 2×10^{-8} ampere, equivalent to a change in indicated background pressure of about 2×10^{-7} torr.

The increase in background current was assumed to be due to a photoelectric emission current from the gage collector as a result of the heated filament. Outgassing of the gage elements permitted the original background current to be recovered.

The value of 2×10^{-8} ampere for the collector photoelectric current is in close agreement with a value of 7.5×10^{-9} ampere calculated for the RG-75 gage. The photoelectric current due to cesium is generally approximately three to four decades greater than the conventional X-ray current observed in the particular gage in question.

Errors

In the process of data recording, cesium temperatures were recorded to $\pm0.1^{\circ}$ C, while the ion and electron currents were measured to ±1 percent. The other gage parameters (voltages and currents) were measured to ±5 percent.

Reference 7 shows an accuracy of ±1.0 percent for the vapor-pressure equation. The error of $\pm0.1^{\circ}$ C in the temperature measurement would add an additional error of ±0.7 percent in the temperature range of interest, so that a total error in the measurement of cesium pressure of less than ±2.0 percent is assumed. In fact, measurements indicated a general decay of sensitivity with a decrease in cesium purity. Since vapor pressures for impure cesium were not known, quantitative results could not be given other than for the case of assured cesium purity. Only when cesium purity was known to be high, were measurements accepted.

Several sources of error were recognized and analyzed. Variations in envelope temperature caused transpiration errors. So that this problem might be eliminated, the gage was tested only at temperatures found under normal operating conditions.

Changes in tubulation temperature caused, in turn, changes in background pressure. A simple calculation showed that these temperature effects, with consideration of geometry and the range of temperatures involved, should not exceed ±0.5 percent. If this effect had been sizable, the data points of figure 5 would have fallen on lines having a slope greater than unity. Since such an effect was not noted, the estimate of the maximum error involved was assumed to be realistic.

A retrospective analysis of the data indicated a most probable value for cesium sensitivity of 13.7 times the air sensitivity with a probable error of 14 percent for a single observation.

CONCLUDING REMARKS

An attempt has been made to predict the ionization gage sensitivity to cesium with a method similar to that of references 3 and 4. With this method and the cesium ionization data of references 9 to 11, the gage sensitivity to cesium was calculated to be 0.85 to 1.85 times the air sensitivity. Although the calculation is accurate to, at best, a factor of about 2, the observed and the calculated values differ by a factor of about 10. There are two possible reasons for this difference: the data of reference 11, as interpreted in reference 12, are incorrect, or a second mechanism of ionization results in the radically high cesium gage sensitivity. Although the latter conclusion is preferred herein, the former is drawn in reference 13.

A possible mechanism to yield the observed cesium sensitivity might be ionization by bombardment of the vapor with photons from the heated filament. If this were the case, the same mechanism should also be exhibited with the other alkali metals, so that the ionization gage would have extremely high sensitivities to these vapors.

SUMMARY OF RESULTS

A study was conducted to determine the effects of cesium vapor on Bayard-Alpert ionization gages at pressures less than 10^{-5} torr. The results of this study can be roughly divided into three main parts. First, a technique described herein, was developed by which similar studies with condensable vapors could be conducted. With minor modification, this technique should be useful with a variety of condensable materials in studies on devices similar to the ionization gage.

Secondly, measurements were made on the ionization gage that indicated that the ionization gage sensitivity to cesium is 13.7 times the air sensitivity with a probable error of 14 percent.

Finally, a number of effects of cesium on the gage were observed, all traceable to the condensable and conductive nature of cesium. These effects included a time constant of the order of hours in response to changes in cesium pressure, photoelectric effects, and temporary gage failure when cesium plating between the electrode lead became excessive.

Lewis Research Center
National Aeronautics and Space Administration
Cleveland, Ohio, November 21, 1963

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TABLE I. - TYPICAL SENSITIVITY DATA FOR RG-75 IONIZATION GAGES IN CESIUM VAPOR ENVIRONMENT

[Background collector current, 4.6×10^{-8} amp; electron current, 10 ma; grid voltage, 180 v; filament voltage, 30 v; collector voltage, 0.]

Cesium temperature, °C	Cesium vapor pressure, torr	Collector current, amp	Collector current less background, amp	Gage sensitivity, $torr^{-1}$	Sensitivity, relative to air
-20	1×10^{-9}	4.6×10^{-8}	0	(a)	(a)
.3	8.3×10^{-8}	1.9×10^{-7}	1.4×10^{-7}	169	16.9
4.5	1.4×10^{-7}	2.2×10^{-7}	1.7×10^{-7}	121	12.1
10.0	2.6×10^{-7}	4.0×10^{-7}	3.5×10^{-7}	135	13.5
12.8	3.5×10^{-7}	4.2×10^{-7}	3.7×10^{-7}	106	10.6
18.3	6.5×10^{-7}	1.8×10^{-6}	1.7×10^{-6}	262	26.2

[a]Background current.

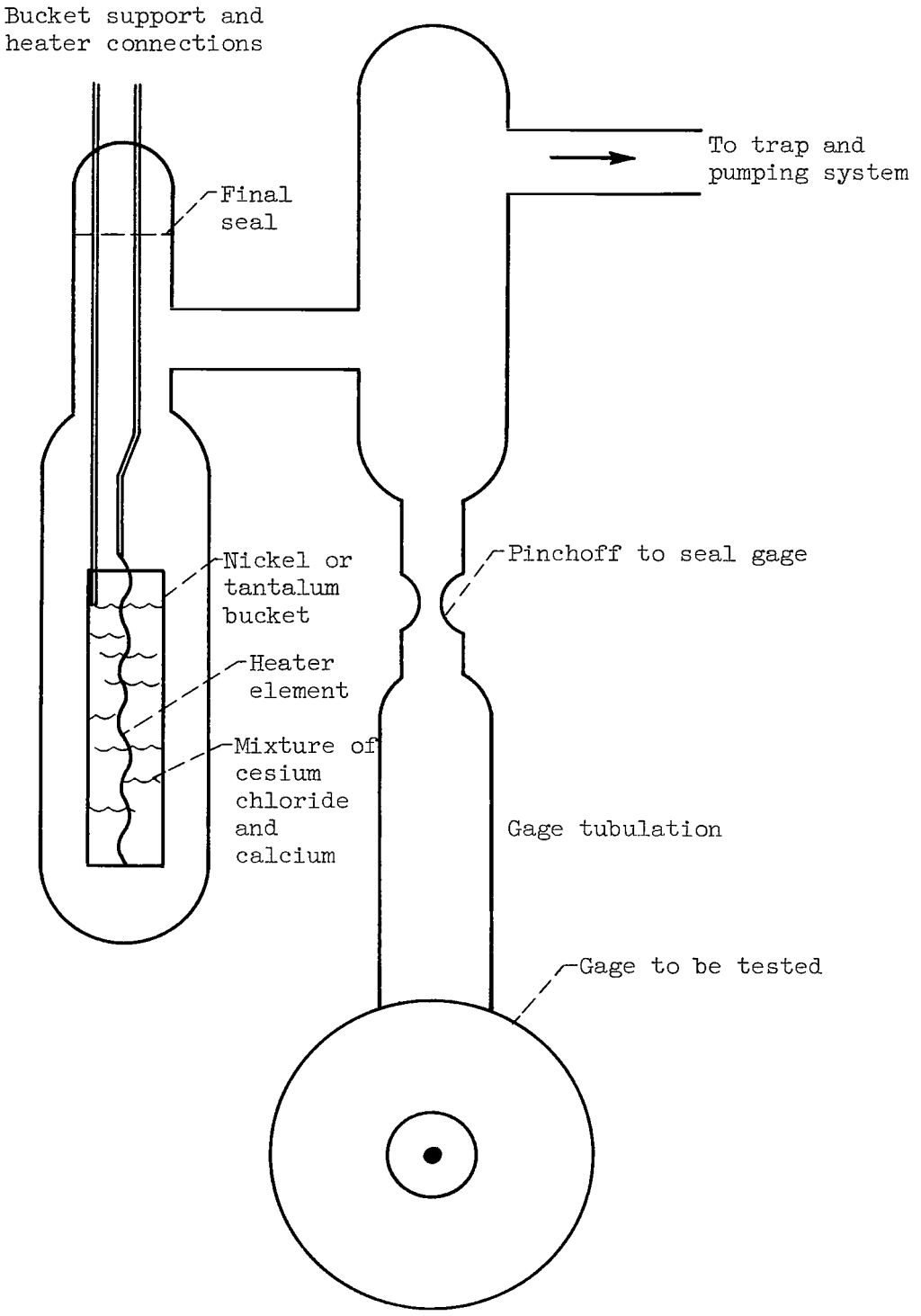

Figure 1. - Schematic drawing of apparatus to prepare ionization gages for testing in cesium vapor environment. All-glass system (bakeable to 350° C).

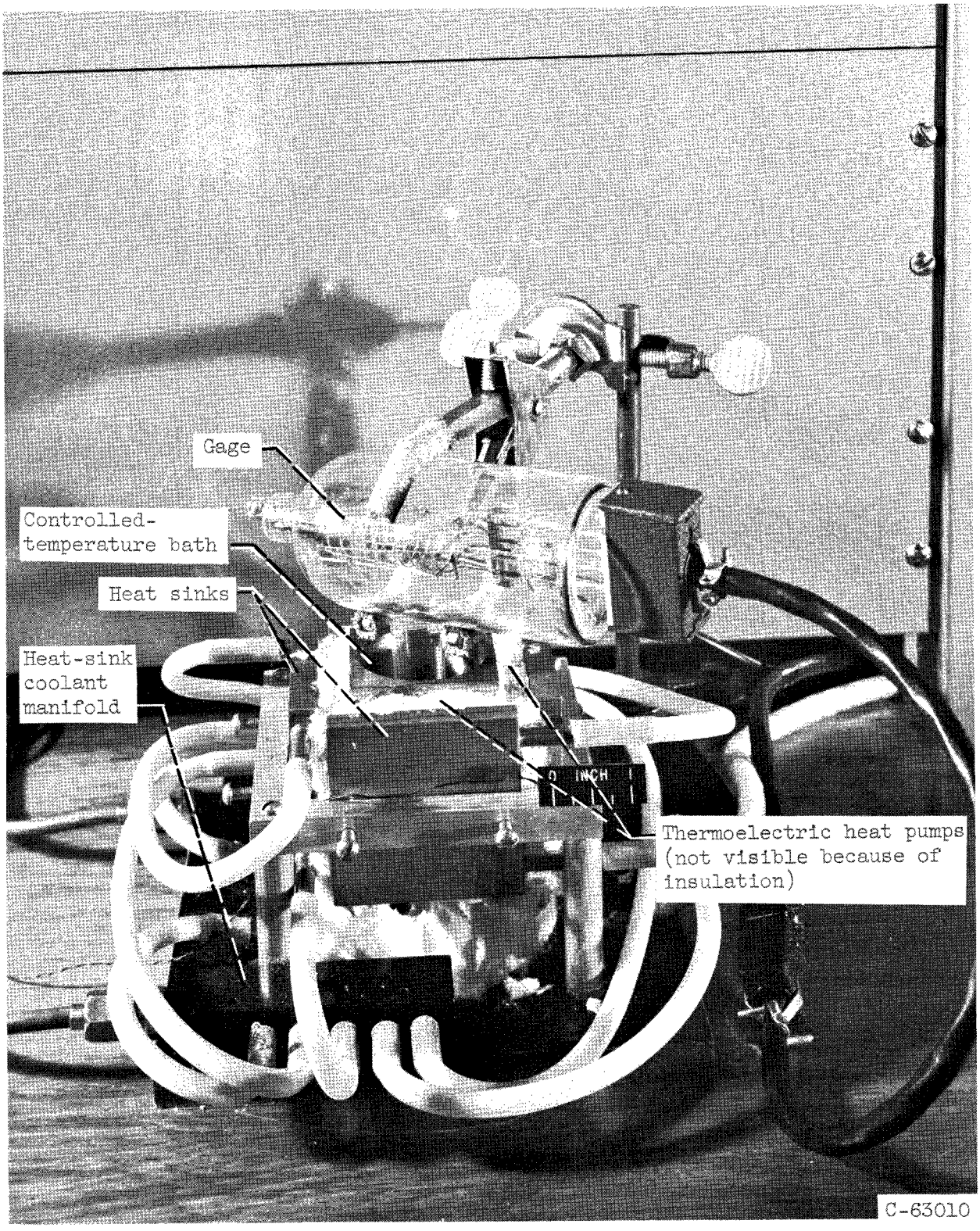

Figure 2. - Gage mounted in test apparatus.

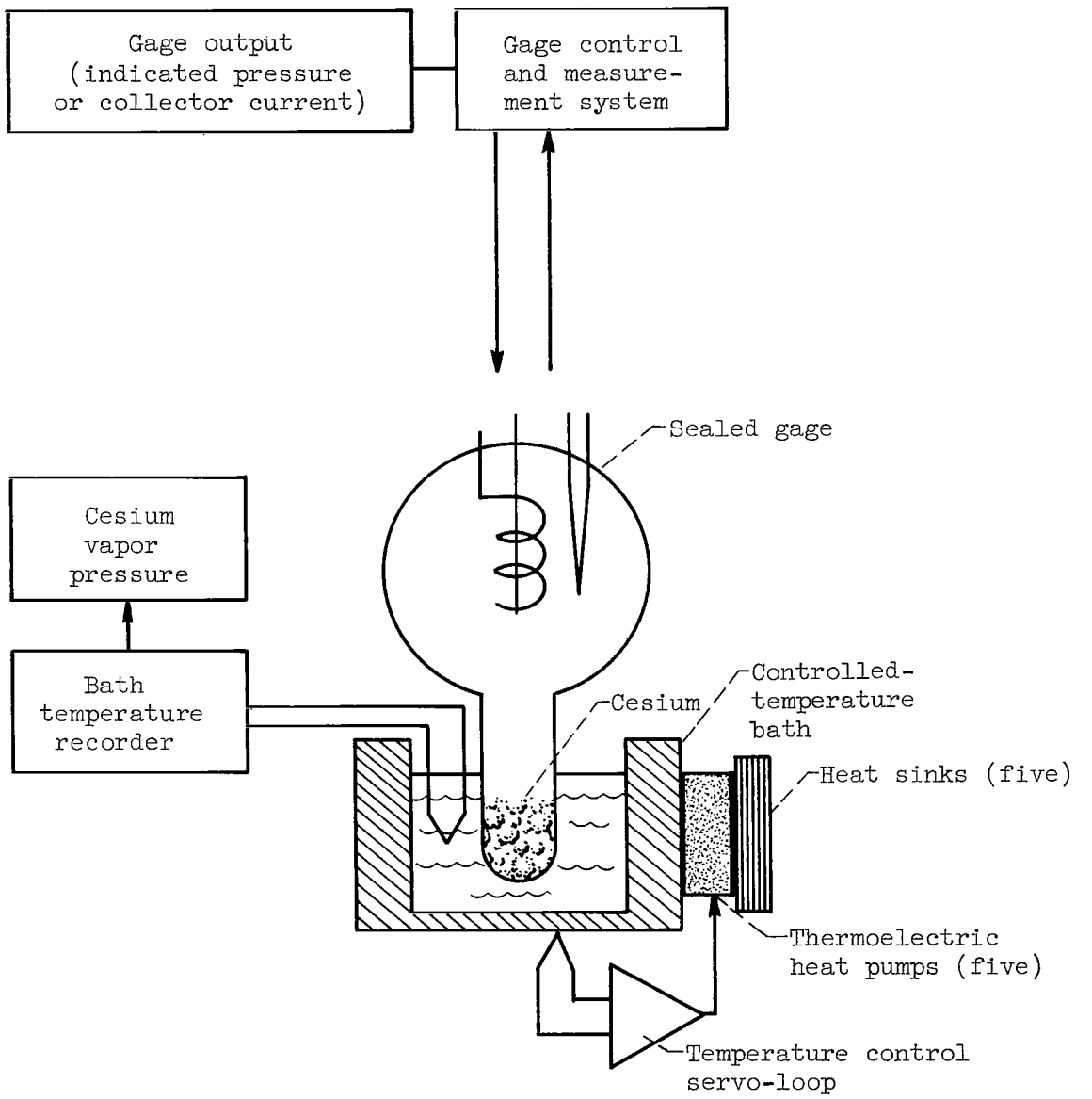

Figure 3. - Ionization gage testing apparatus for determination of gage sensitivity to cesium vapor. Cesium temperature less than gage temperature.

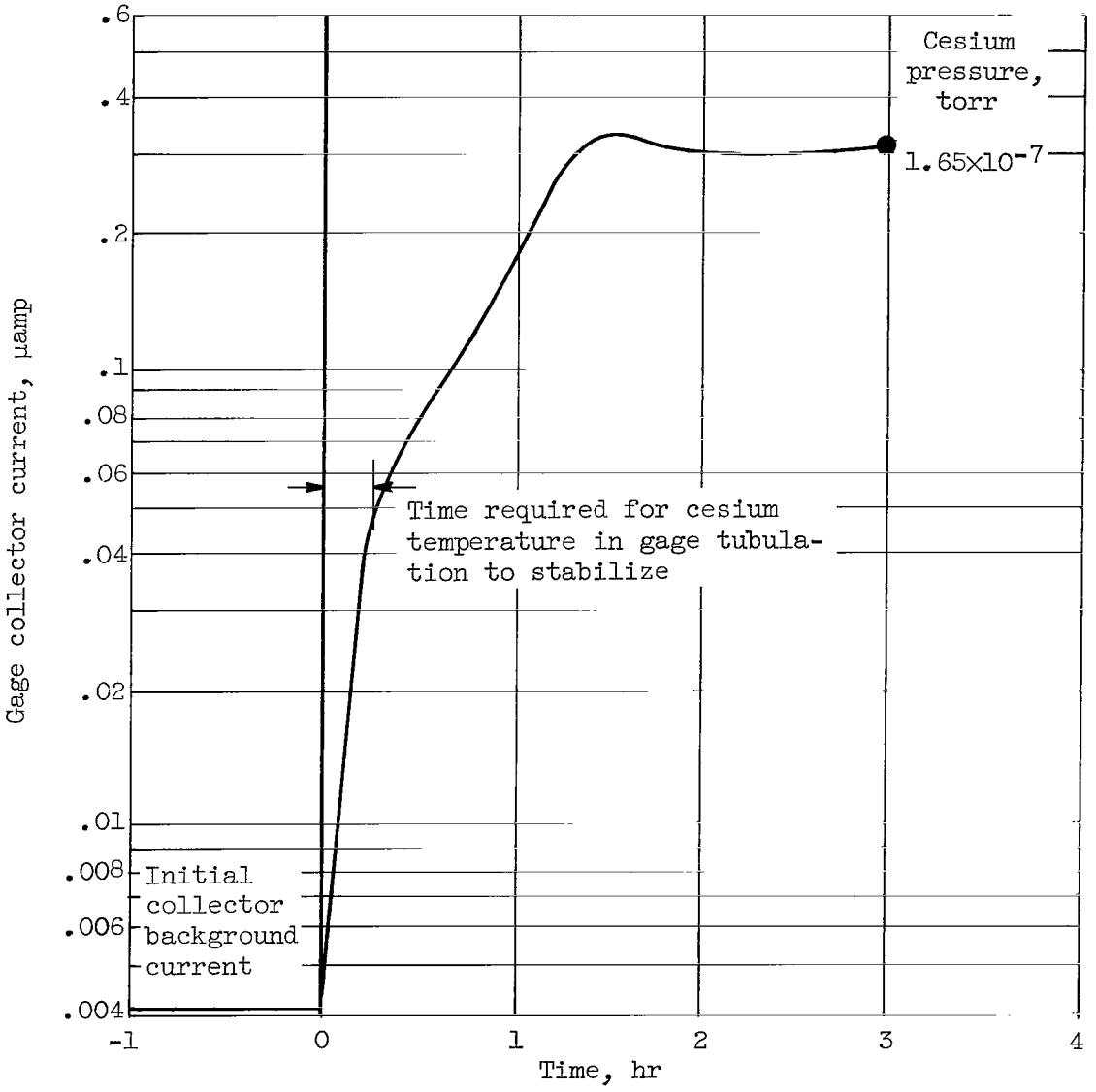

Figure 4. - Typical response of RG-75 ionization gage to step change in cesium pressure.

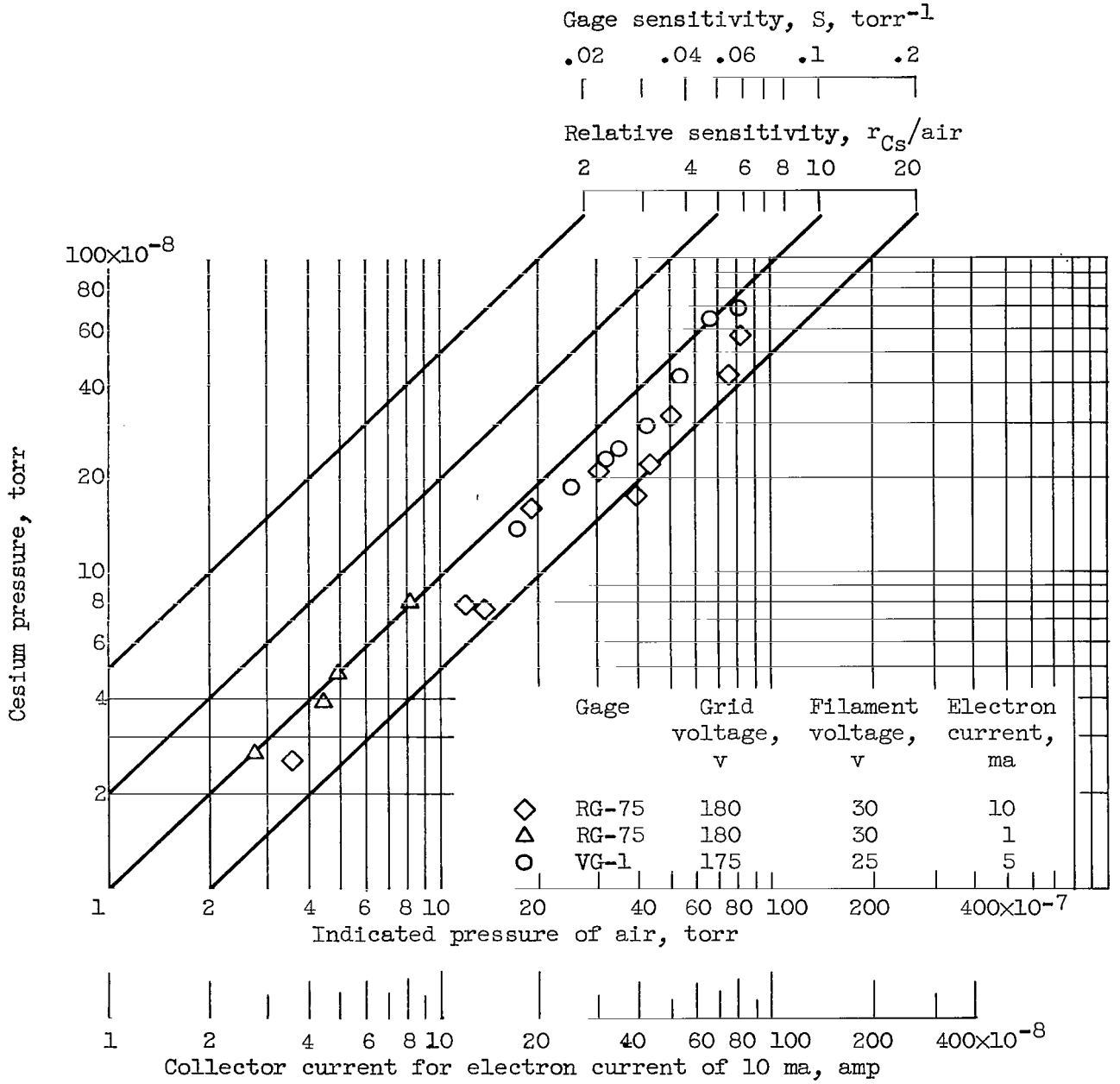

Figure 5. - Ionization gage indicated pressure as function of cesium vapor pressure. Voltages measured relative to collector.